Pilot-Scale Testing of an Integrated Circuit for the Extraction of Rare Earth Minerals and Elements from Coal and Coal Byproducts Using Advanced Separation Technologies

PRINCIPAL INVESTIGATOR:  
Dr. Rick Honaker  
University of Kentucky

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NETL Program Manager: Charles Miller

2019 Annual Project Review Meeting  
Crosscutting Research, Rare Earth Elements, Gasification Systems, and Transformative Power Generation  
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Project Objectives

• Develop, design and demonstrate a ¼-tph pilot-scale processing system for the recovery of high-value rare earth elements (REEs) from coal and coal byproducts.

Integration of physical and chemical separation processes as needed;

Production of concentrates with purity levels of at least 2% total REEs;

Technical and economic feasibility.
Project Team

University of Kentucky Research Foundation

Principal Investigator
(Rick Honaker)

Project Management Committee
Academic Members
- University of Kentucky
- Virginia Tech
- West Virginia University

Corporate Members
- Blackhawk Mining, LLC
- Alliance Coal
- Minerals Refining Co.
- Mineral Separation Technologies

Rick Honaker (UKY)
John Groppo & Josh Werner (UKY)
Roe-Hoan Yoon (VT)
Gerald Luttrell (VT)
Aaron Noble (VT)
Qingqing Huang & John Herbst (WVU)

BP2-Task 1: Project Management and Planning
BP2-Task 2: Site Host Agreements
BP2-Task 6: Bidding & Procurement
BP2-Task 5: Site Rehabilitation
BP2-Task 8: Installation & Assembly
BP2-Task 10: Startup & Shakedown
BP2-Task 9: Systems Safety Analysis & Training
BP2-Task 1: Project Management and Planning
BP2-Task 8: Detailed Systems Engineering
BP2-Task 7: Fabrication & Construction
BP2-Task 8: Installation & Assembly
BP2-Task 10: Startup & Shakedown

BP3-Task 1: Project Management and Planning
BP3-Task 3: Feedstock Collection & Preparation
BP3-Task 5: Test Plan Revision
BP3-Task 12: Sample Analyses
BP3-Task 16: Phase 2 Summary Report
BP3-Task 8: Provide SpR Samples
BP3-Task 6: Detailed Parametric Testing
BP3-Task 7: Optimization & Validation
BP3-Task 15: Decommissioning & Disposition
BP3-Task 1: Project Management and Planning
BP3-Task 16: Phase 2 Summary Report
BP3-Task 4: Exploratory Testing
BP3-Task 9: Secondary Feedstock Testing
BP3-Task 10: Plant Relocation & Recommissioning
BP3-Task 11: Modeling & Simulation
BP3-Task 14: Commercialization Analysis
BP3-Task 2: Environmental Monitoring & Management
Feed Type A1 (Plant Coarse Reject)
Feed Type A2 (Refuse Pile)
Feed Type B1 (Plant Fine Reject)
Feed Type B2 (Pond Fines)
Feed Type C1 (Natural Leachate)
Feed Type C2 (Pile Leachate)
Feed Type C3 (Leach Pad)

Process Flowsheet

COAL PRODUCT

Sorter → Crush/Grind → Coal Recovery → Alkali Rejection

REM Recovery → Roast/Crack

Leach/Ion Exchange

Reduction/pH Control → Solvent Extraction → Oxalic Acid Precipitation

BULK REE CONCENTRATE

SPENT WASTE
REE Pilot Plant Location:
Providence, Kentucky
Process Modules

1.00 – Preconcentration (Mine Site)
2.00 – Size Reduction/Liberation
3.00 – Physical Separation
4.00 – Acid Leaching
5.00 – Solvent Extraction & Precipitation
6.00 – Chemical Storage
7.00 – Rare Earth Mineral Concentration
REE Pilot Plant
(https://m.youtube.com/watch?v=jR70j-MzWNE)
PLC Control System
Analytical Lab

ICP-OES & Fume Hood

Deionized Water System
REE Preconcentration

- Belt Sampler
- Reject Conveyor (+ 50 mm/-6 mm)
- Oversize/Fines Pile
- Vibrating Screen
- Sized Product (50x6 mm)
- Electronic X-Ray Sorter
- Product & Reject Conveyors
- Middling Product
- Coarse Waste
- Coarse Feedstock

Feedstock → Oversize/Fines Pile → Reject Conveyor → Vibrating Screen → Sized Product → Electronic X-Ray Sorter → Product & Reject Conveyors → Middling Product → Coarse Waste → Coarse Feedstock
REEs concentrate in the middle density fractions of high rank coals... which allows the potential of preconcentration based on particle density.

REE Preconcentration
Technology Description

Feed

- Pyrite
- Coal
- Coal-Rock Middling
- Rock
- Magnified Image

Drilet™ Sorter

Clean

- Coal
- Low-Ash Middling

Reject

- Pyrite
- High-Ash Middling
- Rock
Technology Description

- **Feed Conveyor (Low Speed)**
- **Scanning Conveyor (High Speed)**
- **X-Ray Source**
- **Compressed Air Jets**
- **Electronics & Controls**
- **X-Ray Detector**
- **Coal**
- **Rock**
- **Reject**
- **Clean**
Mobile REE Sorter Performance

<table>
<thead>
<tr>
<th>REE Elements</th>
<th>Pass (ppm)</th>
<th>Eject (ppm)</th>
<th>CR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ash (%)</td>
<td>89.6</td>
<td>78.1</td>
<td>0.90</td>
</tr>
<tr>
<td>TREE (ppm)</td>
<td>312</td>
<td>438</td>
<td>1.29</td>
</tr>
<tr>
<td>HREE (ppm)</td>
<td>44</td>
<td>61</td>
<td>1.28</td>
</tr>
<tr>
<td>LREE (ppm)</td>
<td>268</td>
<td>377</td>
<td>1.29</td>
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</table>
Leaching Circuit

- **H₂SO₄** (Sulfuric Acid)
- **Water**
- **Solids From Roasting**
- **Leach Tank 1, 2, 3**
- **Leach Thickener**
- **Leach Filter Feed Tank**
- **Leach Filter**
- **PLS to Solvent Extraction**

**Treatment Chemicals**
- **Recycled Raffinate**
- **Water Treatment Tank**
- **Water Clarifier**
- **PG Pump**
- **Sludge Filter**
- **Solid Waste (Precipitate)**
- **To Water Discharge**
- **Solid Waste (Particulate)**
- **To Waste Disposal**

* PLS = Pregnant Leach Solution
Pilot Plant Leach Circuit
Minimum Acid and Base Cost Assessment

- Cost of $H_2SO_4$ and NaOH needed to reduce pH from 7.0 and increase to a value of 8.0.
- PLS = Pregnant Leach Solution

- Assumes 100% recovery of REEs;
- Liquid:Solid ratio = 5:1;
- Consumption by solids not included.
Pre-Leach Roasting

• Roasting at an optimum temperature of 500 – 750 °C causes:
  • Decomposition of rare earth minerals in the presence of activating reactants
  • Decomposition of clays
• Fuel is provided by the presence of carbonaceous material.
• Advantages include:
  • Improved REE recovery
  • Reduced acid consumption
  • Increased leaching kinetics
  • Reduced contaminant content in PLS.
Pre-Leach Roasting

WK No. 13 Coarse Refuse

Pocahontas No. 3 Coarse Refuse
Laboratory Column Heap Leach Tests
Heap Leach PLS: SX Feed

West Kentucky No. 13 Refuse Heap Leach

Elemental Analysis

<table>
<thead>
<tr>
<th>Element</th>
<th>PPM</th>
</tr>
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<tbody>
<tr>
<td>Sc</td>
<td>0.78</td>
</tr>
<tr>
<td>Y</td>
<td>3.90</td>
</tr>
<tr>
<td>La</td>
<td>0.31</td>
</tr>
<tr>
<td>Ce</td>
<td>2.25</td>
</tr>
<tr>
<td>Pr</td>
<td>0.88</td>
</tr>
<tr>
<td>Nd</td>
<td>1.09</td>
</tr>
<tr>
<td>Sm</td>
<td>0.62</td>
</tr>
<tr>
<td>Eu</td>
<td>0.19</td>
</tr>
<tr>
<td>Gd</td>
<td>2.65</td>
</tr>
<tr>
<td>Tb</td>
<td>0.29</td>
</tr>
<tr>
<td>Dy</td>
<td>0.95</td>
</tr>
<tr>
<td>Ho</td>
<td>&lt; 0.003</td>
</tr>
<tr>
<td>Er</td>
<td>0.01</td>
</tr>
<tr>
<td>Tm</td>
<td>0.09</td>
</tr>
<tr>
<td>Yb</td>
<td>0.31</td>
</tr>
<tr>
<td>Lu</td>
<td>0.14</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>14.45</strong></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Element</th>
<th>PPM</th>
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</thead>
<tbody>
<tr>
<td>Th</td>
<td>&lt;0.003</td>
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<tr>
<td>U</td>
<td>1.53</td>
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<tr>
<td>Fe</td>
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<tr>
<td>Al</td>
<td>1467</td>
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<tr>
<td>Ca</td>
<td>459</td>
</tr>
<tr>
<td>Mg</td>
<td>572</td>
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<tr>
<td>Mn</td>
<td>77.6</td>
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Total 14.45
SX Rougher Circuit
## SX Circuit Conditions

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
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<tbody>
<tr>
<td>Feed Rate</td>
<td>0.5 gpm</td>
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<tr>
<td>Organic : Aqueous</td>
<td>1 : 1</td>
</tr>
<tr>
<td>Solvent</td>
<td>Orform</td>
</tr>
<tr>
<td>Extractant</td>
<td>DEHPA</td>
</tr>
<tr>
<td>Extractant Dosage</td>
<td>5% by volume</td>
</tr>
<tr>
<td>Phase Modifier</td>
<td>TBP</td>
</tr>
<tr>
<td>Modifier Dosage</td>
<td>10% by volume</td>
</tr>
<tr>
<td>Feed pH</td>
<td>2.0</td>
</tr>
<tr>
<td>Reducing Agent</td>
<td>Ascorbic Acid</td>
</tr>
<tr>
<td>Strip Solution</td>
<td>6M HCl</td>
</tr>
<tr>
<td>Scrub Solution</td>
<td>0.5M HCl</td>
</tr>
</tbody>
</table>
Rougher Circuit Start-up Performance

SX Feed pH changed to 2.1

Stripping flow rate 6 RPM
Stripping flow rate 10 RPM
Stripping flow rate 18 RPM
Stripping flow rate 20 RPM
Feed to REO Products

Coarse Refuse

RE Oxalate

RE Oxide
# SX Circuit REO Concentrates

<table>
<thead>
<tr>
<th>Rare Earth Element</th>
<th>REO Concentration (%)</th>
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<tbody>
<tr>
<td></td>
<td>27-Nov</td>
</tr>
<tr>
<td>Sc</td>
<td>0.02</td>
</tr>
<tr>
<td>Y</td>
<td>17.49</td>
</tr>
<tr>
<td>La</td>
<td>0.23</td>
</tr>
<tr>
<td>Ce</td>
<td>6.94</td>
</tr>
<tr>
<td>Pr</td>
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<tr>
<td>Nd</td>
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<tr>
<td>Sm</td>
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<tr>
<td>Eu</td>
<td>3.69</td>
</tr>
<tr>
<td>Gd</td>
<td>18.00</td>
</tr>
<tr>
<td>Tb</td>
<td>2.65</td>
</tr>
<tr>
<td>Dy</td>
<td>10.31</td>
</tr>
<tr>
<td>Ho</td>
<td>1.38</td>
</tr>
<tr>
<td>Er</td>
<td>1.65</td>
</tr>
<tr>
<td>Tm</td>
<td>0.00</td>
</tr>
<tr>
<td>Yb</td>
<td>0.10</td>
</tr>
<tr>
<td>Lu</td>
<td>0.00</td>
</tr>
<tr>
<td>Total</td>
<td>93.02</td>
</tr>
</tbody>
</table>
Conclusions:
- Water reduction process is uneconomical.
- Preconcentration of the REEs in the PLC to SX is required.
Summary & Conclusions

• A pilot plant that integrates physical and chemical separation processes to recovery REEs from coal-based sources has been designed and installed. Testing is on-going.

• Roasting provides significant REE recovery and chemical reduction benefits.

• The current SX circuit has the ability to produce high quality REO concentrates; however, a reduction in chemical costs is required.

• The low REE contents in coal-based sources limits the amount of acid that can be used to improve REE recovery.

• Reduction of PLS solution prior to SX is uneconomical.

• Preconcentration of REEs in the PLS to around 500 ppm prior to SX is required.
Next Steps

• Roasting
  • Determining the impact of activating agents
  • Installation of continuous roaster
  • Optimization

• Tank Leaching
  • pH 2 leaching no control
  • pH 2 leaching with control
  • Heap leaching

• Selective Precipitation
  • Tests with and without reduction of Fe$^{2+}$ at lab and pilot plant scale

• Modify circuitry
Thank You...